

Article

## Photo Stabilizing Efficiency Of Poly(methyl methacrylate) Based On Metal Ion Complexes Of Saccharine

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### ABSTRACT

The photostabilization of poly(methyl methacrylate) (PMMA) films having saccharine complexes of Zn(II), Cd(II), Hg(II), Fe(III), Ni(II) and Cu(II) complexes was investigated. The production of PMMA films containing such complexes (0.5% by weight) was carried out by the casting method using THF. The photostabilization activities of the compounds were determined by monitoring the carbonyl and hydroxyl index with irradiation time. Evaluation of stabilizing efficiency of PMMA by ultra-violet spectroscopy to investigate photodecomposition rate constant ( $kd$ ) for PMMA films in the absence and presence of saccharine complexes. The changes in the viscosity average molecular weight of PMMA with irradiation time were evaluated. The rate of photostabilization for PMMA in the presence of the additives was found to follow the order Ni(sac) > Cu(sac) > Zn(sac) > Cd(sac) > Hg(sac) > Fe(sac). Depending on the structure of the additive, such as a peroxide decomposer, UV absorption or a radical scavenger for the photostabilizer.

**Keywords:** Photodegradation, UV stabilizers, PMMA, Saccharine.

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### INTRODUCTION

PMMA is considered one of the commercially available polymers<sup>1</sup> It is widely used in many applications due to its distinctive combination of excellent optical properties such as thermal stability, easy formation, some good mechanical properties, and weather resistance.<sup>2</sup> However, this polymer has some defects such as embrittlement and low chemical resistance that can be removed by chemical, or physical modification poly(methyl methacrylate) is one of the oldest and most famous types of polymers and was seen as an alternative to glass in a variety of applications and is currently used in glass applications as it is characterized by hardness and hardness. When it is in its natural form, it becomes a colorless transparent material that allows the transfer of A large amount of visible light by (92%) and visible light by (4%) is reflected on the surface<sup>3</sup>. Plastics replace glass, metals, and wood in many modern applications. 4. Unlike conventional materials,

plastic has superior properties and special performance.<sup>5</sup> Certain materials can degrade when exposed to ultraviolet radiation (UV). UV absorbers (UVAs) are chemical compounds that can shield materials from the harmful effects of UV light. UVA can be integrated into a material to protect it from UV radiation, or a UVA-containing composition can be applied to a UV-sensitive substrate to shield it from UV radiation. UV light stabilizers are found in various products, including films, polymers, and cosmetics. UV solar light has enough energy to cause photochemical deterioration of materials in outdoor applications via chemical reactions. Antioxidants, heat, and light stabilizers can be added to polymers to prevent deterioration<sup>6</sup>. UV stabilizers protect polymeric materials from photodegradation and photocrosslinking generated by artificial light and ultraviolet sunshine. Organic and inorganic moieties are found in a variety of UV light stabilizers. Because of their unequal distribution within plastic substrates, inorganic UV stabilizers containing chromic, titanium, iron oxides, and carbon black are frequently incompatible with the polymer matrix. The UV-final stabilizer's effects depend on the concentration and particle size<sup>7</sup>. Almost all synthetic polymers require stabilization against the adverse effect. With the development of synthetic resins, it became necessary to look for ways and means to prevent, or at least reduce, the damage caused by environmental parameters such as light, air and heat. This can be achieved by adding special chemicals, light stabilizers or UV stabilizers that must be adjusted to the nature of the resin and the specific application considered. The photostabilization of polymers may be achieved in many ways. The following stabilizing systems have been developed, which depend on the action of the stabilizer: (a) light screeners, (b) UV absorbers, (c) excited state quenchers, (d) peroxide decomposers and (e) free radical scavengers, of these it is generally believed that types

(c)–(e) are the most effective. Most stabilizers are believed to be multifunctional in their mode of operation. This view is complicated because photooxidation mechanisms depend on the polymer structure and other variables, such as manufacturing, operation, processing and conditions<sup>8,11</sup>. In a recent study, we became interested in the photostabilization of PMMA using saccharine complexes to investigate the photostabilization of PMMA films.

## Materials and Methods

All reagents were commercially available and used without further purification. Elemental analyses (C, H, N and S) were carried out on an Elementar Eager 300 elemental analyzer. The FT-IR spectra were recorded from KBr pellets in the 400 – 4000  $\text{cm}^{-1}$  [65 FT-IR Perkin Elmer Spectrophotometer]. Magnetic susceptibility data were measured from powder samples of solid material in the 2-300 K temperature range using a SQUIT susceptometer MPMS-7, Quantum Design with a field of 1.0T. The experimental data were corrected for underlying diamagnetism by the use of tabulated Pascal's constants

## Synthesis of metal complexes of saccharin

The starting complexes  $[\text{M}(\text{sac})_2(\text{H}_2\text{O})_4]$  were prepared according to the literature methods reported by Haider *et al.*<sup>12</sup> and Kamenar *et al.*<sup>13</sup>, respectively.

## Films preparation

Commercial PMMA was re-precipitated from THF solution by alcohol several times and finally dried under vacuum at room temperature for 24 h. Fixed concentrations of PMMA solution (1 g/50 ml) in THF were used to prepare polymer films with 80  $\mu\text{m}$  thickness (measured by a micrometer type 2610 A, Germany). The prepared complexes (5% by weight) were added to the films (blank). The films were prepared by evaporation technique at room temperature for 24 h. To

remove the possible residual THF solvent, film samples were dried at room temperature for 3 h under reduced pressure. The films were fixed on stands, especially used for irradiation.

### **Irradiation Experiments: Accelerated Testing Technique**

An accelerated weather-meter Q-panel Laboratory ultraviolet (QUV; Q-Panel Company, Homestead, Florida, USA) tester was used for the irradiation of polymeric films and was supplied by the (Polymer Research Unit, College of Science, Al-Mustansiriyah University). The accelerated weathering tester contains a stainless steel plate with one hole in the back and two others in the front. Each side contains a UV-B 313 fluorescent ultraviolet lamp (40 W) (Q-Panel Company), giving a spectrum within a 290–360 nm range, with 313 nm as the maximum wavelength. The samples of the polymeric film were fixed parallel to the lamps and vertically to ensure that the UV incident radiation was perpendicular to the samples. To ensure that the intensity of incident light is the same on all samples, samples were rotated from time to time

### **Photodegradation Measuring Methods**

#### *Measuring the Photodegradation Rate of Polymer Films Using Infrared Spectrophotometry*

An FTIR Perkin Elmer Spectrum 65 Spectrophotometer (400–4000  $\text{cm}^{-1}$ ) by (JASCO) was used to monitor the degree of photodegradation of polymeric film samples in which hydroxyl group absorption took place at the range (3420 - 3490  $\text{cm}^{-1}$ ) and carbonyl group at the range (1720 - 1740  $\text{cm}^{-1}$ ), The changes in the hydroxyl and carbonyl group's absorption peaks indicated the progress of photodegradation at different irradiation times. The comparison between the FTIR hydroxyl and carbonyl group's absorption peak and the reference peak allows the calculation of the hydroxyl and carbonyl index ( $I_{\text{O-H}}$ ) ( $I_{\text{C=O}}$ ) as shown in Equation (1) <sup>14</sup>.

$$I_s = \frac{A_s}{A_r} \quad (1)$$

Where  $I_s$  = the index of hydroxyl, carbonyl group,  $A_s$  = the absorbance of peak and  $A_r$  = the absorbance of reference peak. The difference between the absorbance at baseline and the top (a top peak—a baseline) is known as the actual absorbance and can be calculated using the baseline method <sup>14,15</sup>.

#### *Determination of Average Molecular Weight (Mv) Using the Viscometry Method*

The polymer's average molecular weight was determined in terms of the viscosity property using the Mark–Houwink relation <sup>16</sup>.

$$[\eta] = K\bar{M}^\alpha \quad (2)$$

Where  $[\eta]$  = the intrinsic viscosity and  $\alpha$  and  $K$  = constants that depend on the polymer-solvent system at a particular temperature. An Ostwald U-Tube Viscometer was used to determine the intrinsic viscosity of the polymer solution (g/100 mL). The pure solvent and polymer solution flow times are  $t_0$  and  $t$ , respectively. Equation (3) was used to calculate the specific viscosity ( $\eta_{\text{sp}}$ ).

$$\eta_{\text{re}} = \frac{t}{t_0} \quad (3)$$

Where  $\eta_{\text{re}}$  = the relative viscosity.

$$\eta_{\text{sp}} = \eta_{\text{re}} - 1 \quad (4)$$

Equation (2) converted the single-point measurements to the intrinsic viscosities.

$$[\eta] = (\sqrt{2}/C)(\eta_{\text{sp}} - \ln\eta_{\text{re}})^{1/2} \quad (5)$$

where  $C$  = the polymer solution concentration (g/100 mL). The molecular weight of the undergirding and polymers can be calculated using equation (5).

## RESULTS

### Evaluation of Stabilizing Efficiency of PMMA by FTIR Spectroscopy

The metal complexes of saccharin and mixed ligand complexes were used as additives for the photostabilization of PMMA films. In order to study the photochemical activity of these additives for the photostabilization of PMMA films, the hydroxyl index was monitored with irradiation time using IR spectrophotometry. The irradiation of PMMA films with UV light of wavelength  $\lambda = 313$  nm led to a clear change in the FTIR spectrum. The appearance of bands in the range (3420 - 3490  $\text{cm}^{-1}$ ) was attributed to the formation hydroxyl group, and at the range (1720 - 1740  $\text{cm}^{-1}$ ) were attributed to the formation carbonyl group<sup>17</sup> Such an absorption band was used to follow the progress of the degradation of the polymer with irradiation and has been calculated as the hydroxyl index. It is fair to link the polymer degradation degree with the growth of the hydroxyl index. However, in Figure 1, the IOH of A<sub>1</sub>, A<sub>2</sub>, A<sub>3</sub>, A<sub>4</sub>, A<sub>5</sub> and A<sub>6</sub> indicated a lower OH growth rate with irradiation time for the PMMA control film without additives. Since the growth of the hydroxyl index with irradiation time was lower than the PMMA blank Figure 1, it was concluded that such additives could be considered photo stabilizers for the PMMA polymer. In principle, a longer induction period indicates an efficient photostabilizer. Therefore, the A<sub>5</sub> is considered the most efficient photo stabilizer, followed by A<sub>6</sub>, A<sub>1</sub>, A<sub>2</sub>, A<sub>3</sub>, and A<sub>4</sub>.

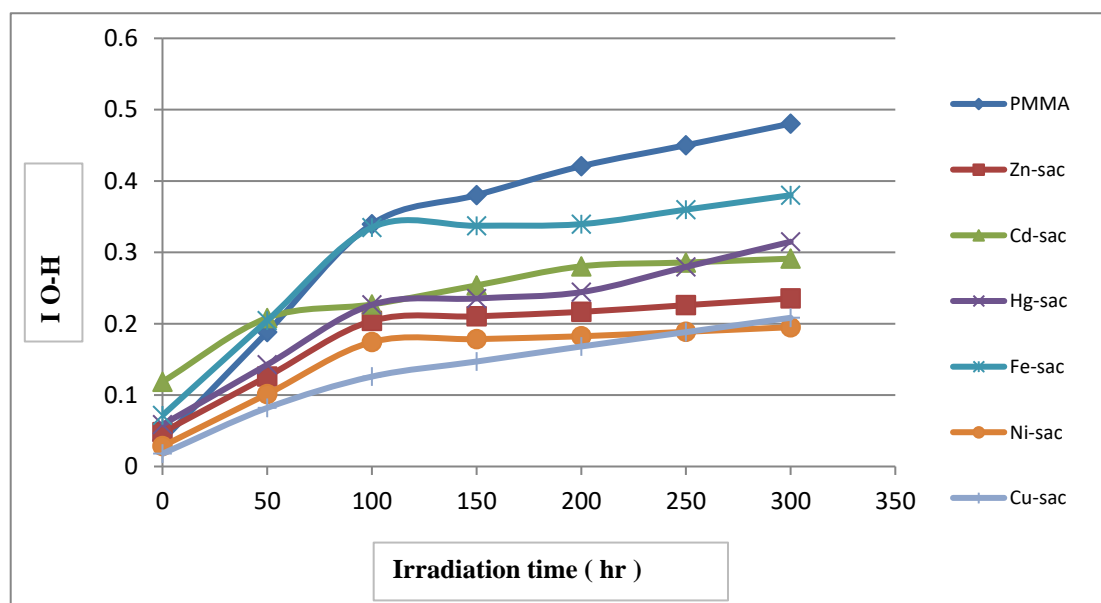


Figure 1. The relationship between the hydroxyl index and irradiation time for PMMA films (80- $\mu\text{m}$  thickness) with different additives (0.5% by weight).

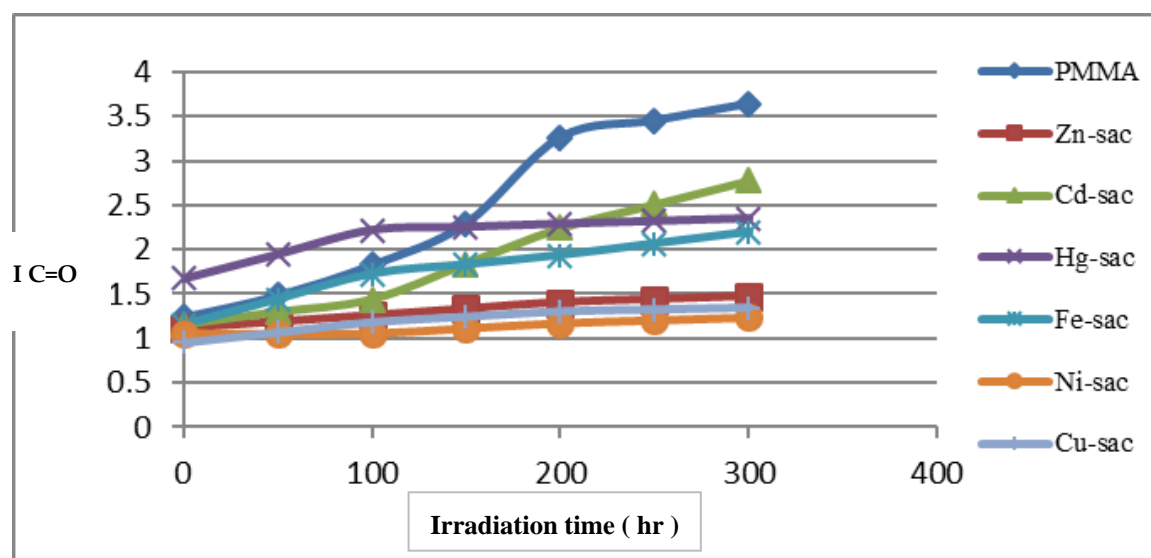


Figure 2. The relationship between the carbonyl index and irradiation time for PMMA films (80- $\mu\text{m}$  thickness) with different additives (0.5% by weight).

#### Evaluation of Stabilizing Efficiency of PMMA by Ultra-Violet Spectroscopy

The effect of saccharine complexes ( $A_1$ ,  $A_2$ ,  $A_3$ ,  $A_4$ ,  $A_5$  and  $A_6$ ) on the PMMA film's photodecomposition was investigated. The PMMA films (80  $\mu\text{m}$  thickness) containing saccharine complexes (0.5% by weight) were irradiated with a UV light ( $\lambda_{\text{max}} = 313 \text{ nm}$ ) for 300 hr. The irradiation led to a clear change in PMMA films and decomposition. The plot of  $\ln(A_t - A_\infty)$  against irradiation time ( $t$ ) gave a straight line. The graphs showed a first-order kinetics in which the slope equals the decomposition rate constant ( $k_d$ ) for PMMA films. The first-order photodecomposition rate constant ( $k_d$ ) for PMMA films in the absence and presence of saccharine complexes (0.5 wt %) is shown in Table (2).

Compound	$K_d (\text{Sec}^{-1}) \times 10^{-3}$
PMMA	8.0
PMMA + $A_1$	3.0
PMMA + $A_2$	4.9
PMMA + $A_3$	6.1
PMMA + $A_4$	5.5
PMMA + $A_5$	2.3
PMMA + $A_6$	3.6

Table 2. The photodecomposition rate constant ( $k_d$ ) for PMMA films on UV irradiation (300 h).

### Variation of PMMA Molecular Weight during Photolysis

Analysis of the relative change in viscosity of average molecular weight (Mv) described by Scott<sup>18</sup> has been shown to provide a test for random string separation; Figure (3) shows the plot (Mv) versus irradiation time of PMMA films with and without 0.5% (w/w) of six selected additives with absorbent light Intensity of  $6.2 \times 10^{-9} \text{ Ein Dm}^{-3} \text{ S}^{-1}$ . We must measure the viscosity of the solution to get an idea of the molecular weight; ultra-viscosity techniques are famous because they are empirically simple<sup>19</sup>. (Mv) measured using equation (2) and THF as a solvent at 25 °C.

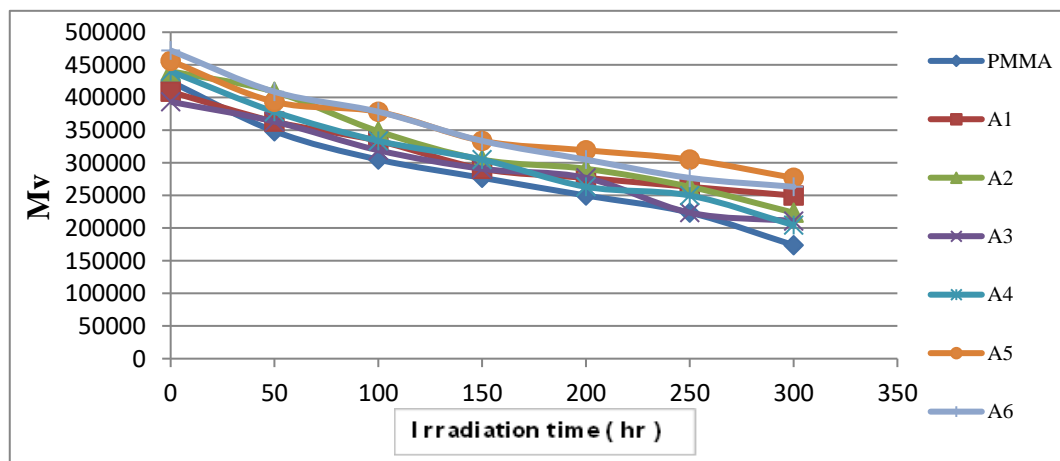


Figure 3. Changes in the viscosity average molecular weight during irradiation for PMMA and modified PMMA films

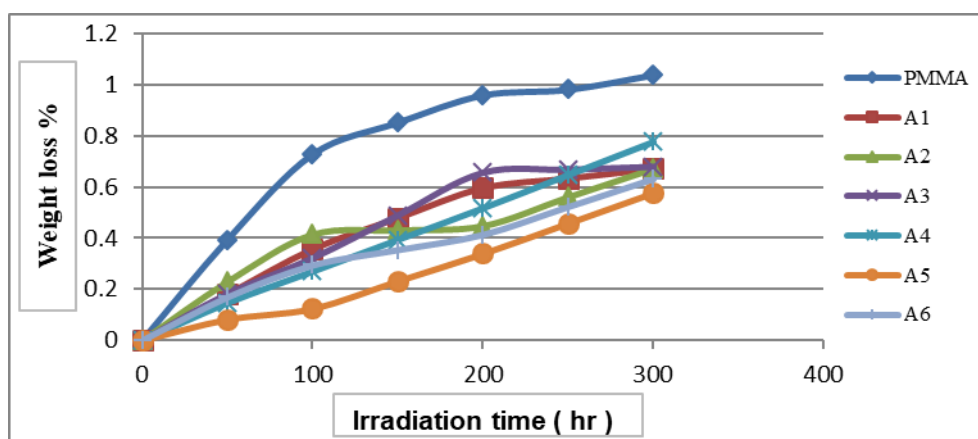
### Determination of the Stabilizing Efficiency by Weight Loss Method

The stabilizing efficiency was determined by measuring the % weight loss of photodegraded PMMA films in the absence and the presence of additives by applying the following equation,

$$\text{Weight loss \%} = \left[ \frac{W1 - W2}{W1} \right] 100 \quad (6)$$

Volatile and low molecular weight products formed on photodegradation of PMMA led to a weight loss phenomenon; the weight loss of PMMA films increased gradually with the increasing degradation time<sup>20,21</sup>; thus, the weight loss percentage as a function of the irradiation time can be a good measure of the degree of degradation and consequently can measure the stabilizing potency of the stabilizer and how long would the stabilizer protect the polymer, The results of weight loss % as a function of irradiation time are represented in Figure (4), the increasing very small with PMMA film modified by additives, The results indicate that all of the five additives have to stabilize effect against photodegradation of PMMA samples and leading to a considerable decrease in % weight loss compared with blank PMMA. The stabilizing efficiency of the investigated photo stabilizers was found to follow this order:

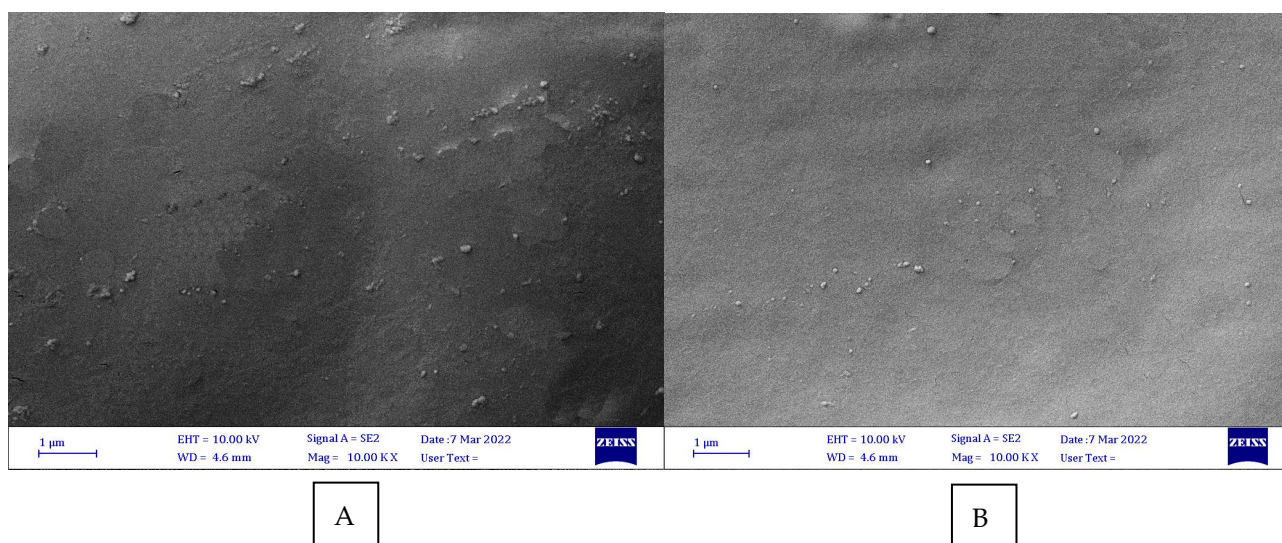
$$\text{Ni(sac)} > \text{Cu(sac)} > \text{Zn(sac)} > \text{Cd(sac)} > \text{Hg(sac)} > \text{Fe(sac)} > \text{PMMA}$$



**Figure 4.** Loss in weight vs. irradiation time for PMMA films containing 0.5% w/w from the additives

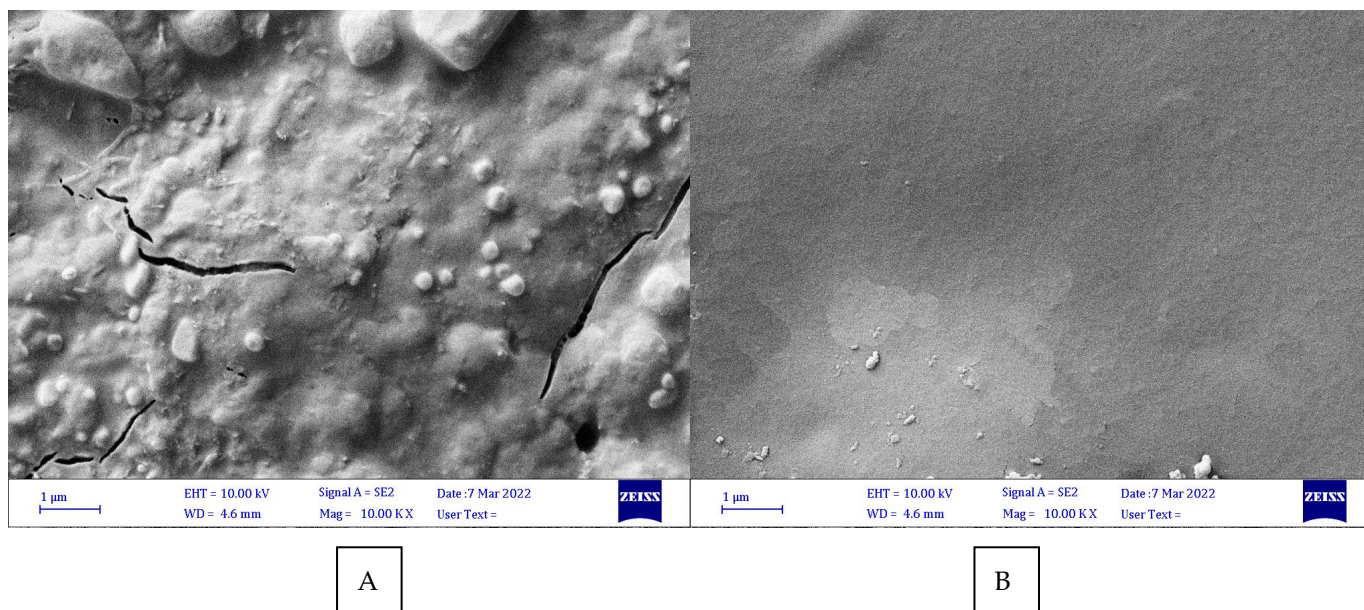
### Surface analysis

The morphological study of the surface of polymers has many advantages. For example, it clearly shows surface irregularities, defects and crystalline state. In addition, it allows monitoring of changes within the surface of the polymeric material as a result of photolysis or stability of polymers when exposed to UV rays in which the decomposition process can occur in the form of chain cleavage<sup>22</sup>; the surface morphology images of the non-irradiated PMMA films in the presence and absence of saccharine complexes are shown in Figure 5. The PMMA film surface was smooth, in which no white spots were detected.



**Figure 5.** Sem images before irradiation of PMMA films blank and with saccharine complexes (0.5% by weight) as additives. A- PMMA blank image before irradiation and B- PMMA+A5 image before irradiation

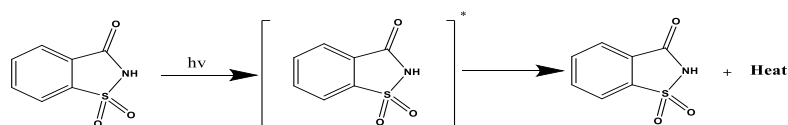
Figure 6 shows the SEM image of PMMA film irradiated for 300 hr in the air. It is clear that the blank PMMA film, irradiated for 300 hr, was full of white spots and grooves due to the photodegradation of polymeric materials and the elimination of ( $\text{CH}_3$  or  $\text{CH}_3\text{COO}$ ). In addition, the morphological image of irradiated PMMA (blank) showed cracks within its surface and color changes<sup>23</sup>. The surface was almost smooth in the presence of saccharine complexes as additives and fewer white spots existed, indicating efficient stabilization effects. Saccharine complexes additives reduce the photodegradation of PMMA film through inhibition elimination of ( $\text{CH}_3$  or  $\text{CH}_3\text{COO}$ ).



**Figure 6.** Sem images for the irradiated (300 hr) PMMA films blank and with saccharine complexes (0.5% by weight) as additives. A- PMMA image after irradiation and B- PMMA+  $A_5$  image after irradiation

## DISCUSSION

The results obtained showed that the efficiency of saccharine complexes as stabilizers for PMMA films could be ordered according to the change in the carbonyl hydroxyl content as a reference for comparison figure(1),(2), where  $Ni(sac) > Cu(sac) > Zn(sac) > Cd(sac) > Hg(sac) > Fe(sac) > PMMA$ . Each additive in the polymeric material plays a role in the photostability of such material. Therefore, various mechanisms for the photostabilization could take place, which were mainly based on the additive chemical structure, the functional groups present, in particular the ones that resonate, such as aromatic and heteroaromatics moieties, and the compatibility of the additive with the polymer matrix. For example, the metal complexes might stabilize PMMA by dissipation energy as heat via direct absorption of the UV radiation<sup>24</sup>. However, the metal's nature will significantly affect the efficiency of the additive as a stabilizer. Both the metal and the ancillary ligands could absorb the UV light<sup>25–27</sup>. Similarly, the aromatic rings in complexes play an essential role in the stabilization mechanism by acting as UV absorbers. The UV light absorption by such additives containing aromatic rings dissipates the UV energy to harmless heat energy (Figure 7). Moreover, the aromatic rings play a role in the resonating structure conjugation of the radical in the peroxide decomposer, which supports this compound as a photostabilizer<sup>28</sup>.

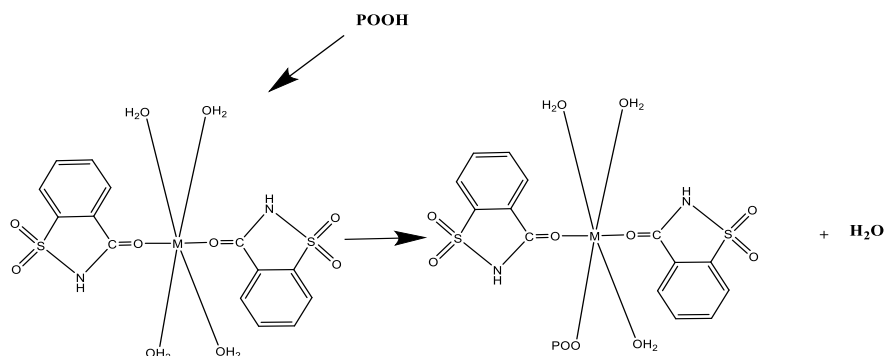


**Figure 7.** Possible photostabilization of aromatic moiety as a UV absorber.

Hydroperoxides play an important role in the degradation of polymeric materials by photooxidation. The use of typical UV stabilizers protecting polymer films through peroxide decomposition has been reported<sup>29</sup>. Metal chelate complexes are PMMA photo stabilizers through a peroxide decomposer and an excited state



quencher. Therefore, it was expected that such complexes could act as peroxide decomposers (Figure 8) <sup>30</sup>.



**Figure 8.** Possible photostabilization of M(sac) complexes as peroxide decomposers

## CONCLUSIONS

The photostabilization of PMMA films of saccharine complexes was studied in which additives act successfully as PMMA film photostabilizers. The order of photo stabilization activity of the additives was Ni(sac) > Cu(sac) > Zn(sac) > Cd(sac) > Hg(sac) > Fe(sac). As a result of the decrease in carbonyl and hydroxyl index for PMMA films, The additives stabilize the PMMA films through different mechanisms, including a peroxide decomposer, a radical scavenger, UV absorption or screening. Such mechanisms support the aim of using saccharine complexes as commercial stabilizers for PMMA. Based on the photostability, the Ni(sac) complex was the most efficient in the photostabilization process.

**Author Contributions:** Noor Sabah Al-Obaidi, Ahmed A. Ahmed Conceptualization of and designed the experiments. Noor Sabah Al-Obaidi performed the experiments and analyzed the data. Ahmed A. Ahmed Ahmed N. Abd provided the funds and revised the paper. Noor Sabah Al-Obaidi wrote the paper. All the authors discussed the results and improved the final text of the paper.

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